PATENT
Docket No. 59098US002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s):	BURTON, Scott et al.	)	Group Art Unit: 1615	
Serial No.:	10/729,114	)	Examiner:	GHALI, Isis A.D.
Filed:	5 December 2003	)	Confirmation No.: 3162	
For:	WOUND DRESSINGS AND METHODS	)		

INFORMATION DISCLOSURE STATEMENT

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

In compliance with the duty imposed by 37 C.F.R. § 1.56, and in accordance with C.F.R. §§ 1.97 *et. seq.*, the materials enclosed herewith are brought to the attention of the Examiner as possibly being of interest in connection with the above-identified patent application. Pursuant to MPEP § 609, the information cited in the present Information Disclosure Statement shall not be construed to be an admission that the information is, or is considered to be, material to patentability. Consideration of each of the documents listed on the attached 1449 form(s) is respectfully requested. Pursuant to the provisions of M.P.E.P. §609, Applicants further request that a copy of the 1449 form(s), marked as being considered and initialed by the Examiner, be returned with the next Official Communication.

Applicants also wish to bring the Examiner's attention to any pending U.S. Application cited in the 1449 form(s) submitted herewith, as well as any documents, Office Actions that may include rejections of similar claims, and any provisional U.S. patent applications referenced in the pending U.S. applications or in their file wrappers. Copies of U.S. Patent Applications 11/550,440 and 11/550,434, which are available on the U.S.P.T.O. Image File Wrapper (IFW) System, are not provided herewith.

Applicants also wish to bring the Examiner's attention to the International Search Report for related international patent application PCT/US2004/040707, a copy of which is provided herewith.

Since this Information Disclosure Statement is submitted after the receipt of an Office Action in the above-identified patent application, Applicants hereby authorize a charge of $180 to Deposit Account No. 13-4895 to cover the fee required under 37 C.F.R. §§1.97(c) and 1.17(p). Please charge any additional fees or credit any overpayment to Deposit Account No. 13-4895.

The Examiner is invited to contact Applicants' Representatives at the below-listed telephone number, if they can be of any assistance during prosecution of the present application.

CERTIFICATE UNDER 37 C.F.R. 1.10:
The undersigned hereby certifies that this paper is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR §1.10 on the date indicated below and is addressed to Mail Stop Amendment, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. Signature: [signature] Name: Margaret S. Willis "Express Mail" mailing label number: EV 201879538 US Date of Deposit: 8 May 2007

May 8, 2007
Date

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INFORMATION DISCLOSURE STATEMENT	**Atty. Docket No.:** 59098US002	**Serial No.:** 10/729,114
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	Information Disclosure Statement mailed:	8 May 2007

U.S. PATENT DOCUMENTS

Examiner Initial	Copy Enclosed	Document Number	Date	Name	Class	Subclass	Filing Date If Appropriate
		3,685,993	08/22/1972	Mukherjee			
		3,761,590	09/25/1973	Fox, Jr.			
		4,728,323	03/01/1988	Matson			
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		2006/0233888 A1	10/19/2006	Burton et al.			
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Examiner Initial	Copy Enclosed	Document Number	Date	Country	Class	Subclass	Translation Yes	Translation No
	X	CN 1169883C	10/06/2004	China			X	
	X	EP 1 601 386 B1	12/07/2006	EP				
	X	WO 2004/080499 A1	09/23/2004	WIPO				
	X	WO 2005/056067 A1	06/23/2005	WIPO				
	X	WO 2005/056069 A1	06/23/2005	WIPO				
	X	WO 2005/056070 A1	06/23/2005	WIPO				

EXAMINER	Date Considered

*Examiner: Initial if citation considered, whether or not citation is in conformance with MPEP 609; Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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	Information Disclosure Statement mailed:	8 May 2007

OTHER DOCUMENTS (Including Authors, Title, Date, Pertinent Papers, etc.)

Examiner Initial	Copy Enclosed	Document Description
	X	ASTM D2857-93 "Standard Practice for Dilute Solution Viscosity of Polymers." Annual Book of ASTM Standards, Vol. 08.01.
	X	International Search Report for PCT Application No. PCT/US2004/040707 (Attorney Docket No. 59098WO003), filed on 12 March 2004; and entitled "WOUND DRESSINGS AND METHODS."
	X	Nesbitt and Sandmann "Solubility Studies of Silver Sulfadiazine." 1977 *J. Pharm. Sci.* 66(4):519-522.
		U.S. Patent Application Serial No. 11/550,434 (Attorney Docket No. 62595US002), filed on 18 October, 2002; and entitled "ANTIMICROBIAL ARTICLES AND METHODS OF MANUFACTURE."
		U.S. Patent Application Serial No. 11/550,440 (Attorney Docket No. 62604US002), filed on 18 October, 2002; and entitled "ANTIMICROBIAL ARTICLES AND METHODS OF MANUFACTURE."

EXAMINER	Date Considered

*Examiner: Initial if citation considered, whether or not citation is in conformance with MPEP 609; Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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权利要求书 1 页 说明书 3 页

[54] 发明名称 纳米级氧化银及其生产工艺

[57] 摘要

一种纳米级氧化银，其平均粒径为 80－100nm，最大粒径与最小粒径之差≤5nm。其制法是将硝酸银加水溶解，在搅拌下逐渐加入浓氨水，配成银氨溶液。另将固体氢氧化钠和保护剂加水搅拌溶解，配成碱溶液。保护剂可以是聚乙烯吡咯烷酮或聚乙烯醇。将银氨溶液在搅拌下逐渐加入到碱溶液中，再充分搅拌后，离心过滤出氧化银，经洗涤和真空干燥后，即得稳定性和分散性好、粒度分布均匀的纳米级氧化银。该方法可用于工业化生产。

1. 一种纳米级氧化银，其特征是其平均粒径为 80～100nm，最大粒径与最小粒径之差为 0～5nm。

2. 一种根据权利要求 1 所述的纳米级氧化银的生产工艺，其特征是将硝酸银配制成水溶液，加入氨水得到银氨溶液，逐渐加入到预先加有保护剂的氢氧化钠溶液中，超细氧化银沉淀即生成，经过滤、洗涤和干燥，得到平均粒径为 80～100nm 的超细氧化银，其最大粒径与最小粒径之差为 0～5nm，所述的保护剂是聚乙烯吡咯烷酮或者是聚乙烯醇。

纳米级氧化银及其生产工艺

本发明涉及纳米级氧化银及其生产工艺。

氧化银（Ag_2O）广泛应用于电子元器件、扣式电池、防腐剂、净化剂、玻璃着色剂和研磨剂、化工催化剂等行业。工业用银中有 30%的银用于氧化银（或硝酸银）生产，其中电子元器件行业所消耗的氧化银约占氧化银总产量的 90%。目前，国内氧化银生产主要采用硝酸银溶液与氢氧化钠溶液反应，得到氧化银沉淀后，经过洗涤、分离和干燥得到氧化银成品。产品的主要技术指标包括氧化银含量、澄清度试验及硝酸不溶物、游离碱、硝酸盐、盐酸不沉物含量，干燥失重等。

主要存在的问题有：

1. 现行产品对颗粒度没有定指标，颗粒度难以达到不同行业的需要。在用银的电子元器件生产中，氧化银一般用来制作氧化银浆，通过球磨方式磨细后与其它物料混合（约需要 7 天左右），再通过丝网印刷到器件基片上。氧化银颗粒度的大小直接影响到所制备的氧化银浆的质量和丝印的费用，进而影响到器件质量。这一行业所要求的氧化银颗粒度越小越好。国内外现行生产工艺难以做到。在化工催化剂、医药防腐剂以及玻璃着色剂等行业所用氧化银同样要求颗粒度赵小越好。

2. 国产氧化银的纯度虽然可以达到国标，但由于现行工艺上的原因，产品颗粒太大，容易包裹杂质，要得到高纯或超纯氧化银难度很大。

本发明的目的是提供一种稳定的、分散性好、粒度分布均匀的纳米级氧化银及其制备方法。

本发明的技术思想：硝酸银溶液直接与氢氧化钠溶液反应时，溶液中的 Ag^+ 浓度过大，生成的氧化银微粒在反应体系中的生长速度过快，同时颗粒之间很容易团聚，导致得到的氧化银产品颗粒过大。若能降低反应时 Ag^+的浓度，使氧化银的生成速度加快而生长速度减慢，同时在氧化银颗粒生成后立即被保护起来．阻止团聚现象的发生，则可得到颗粒度极小的氧化银颗粒产品。

因此本发明的技术方案如下：

一种纳米级氧化银，其平均粒径≤100nm，并且其最大粒径与最小粒径之差≤5nm。

一种纳米级氧化银生产工艺，它是将硝酸银配成水溶液，加入浓氨水得到银氨溶液，逐渐加入到预先加有保护剂的氢氧化钠溶液中，超细氧化银沉淀即生成。经过过滤、洗涤和干燥得到平均粒径为 80-100nm 的超细氧化银。

上述的保护剂可以是聚乙烯吡咯烷酮，或者是聚乙烯醇。

具体的做法是：

将硝酸银50-100份（质量，下同）溶于300份水中，在搅拌下逐渐加入浓氨水100-400份，配成银氨溶液。另将固体氢氧化钠20-40份和0.2-1.0份保护剂加水500份，搅拌溶解，配成碱溶液。保护剂可以是聚乙烯吡咯烷酮或者是聚乙烯醇。将上述银氨溶液在搅拌下逐渐加入到上述碱溶液中，再充分搅拌4-8小时后，离心过滤出氧化银，用水洗涤3次，再用乙醇洗涤3次，在温度低于80℃下真空干燥，即得纳米级氧化银。本发明方法制备的纳米级氧化银粒径为80-100nm，稳定性、分散性好，粒度分布均匀。

采用本发明的生产工艺，得到的氧化银平均粒径为100nm以下，最大粒径与最小粒径之差≤5nm，由于采用了保护剂，因此具有很好的稳定性和分散性。

实施例1

将硝酸银50Kg溶于300L水中，在搅拌下逐渐加入浓氨水100L，配成银氨溶液。另将固体氢氧化钠40Kg和0.2Kg保护剂（聚乙烯吡咯烷酮，简称PVP，分子量为30000）加水500L，搅拌溶解，配成碱溶液。将银氨溶液在搅拌下逐渐加入到碱溶液中，再充分搅拌4h后，离心过滤出氧化银，用水洗涤3次，再用乙醇洗涤3次，在温度为80℃真空干燥，即得纳米级氧化银。本实施例所生产的纳米级氧化银平均粒径为88nm，最大粒径与最小粒径之差≤5nm。

实施例2

将硝酸银100Kg溶于300L水中，在搅拌下逐渐加入浓氨水100L，配成银氨溶液。另将固体氢氧化钠25Kg和1.0Kg保护剂（聚乙烯吡咯烷酮，分子量为30000）加水500L，搅拌溶解，配成碱溶液。将银氨溶液在搅拌下逐渐加入到碱溶液中，再充分搅拌8h后，离心过滤出氧化银，用水洗涤3次，再用乙醇洗涤3次，在温度为80℃真空干燥，即得纳米级氧化银。本实施例所生产的纳米级氧化银平均粒径为100nm，最大粒径与最小粒径之差≤5nm。

实施例3

将硝酸银100Kg溶于300L水中，在搅拌下逐渐加入浓氨水400L，配成银氨溶液。另将固体氢氧化钠40Kg和1.0Kg保护剂（聚乙烯吡咯烷酮，分子量为30000）加水500L，搅拌溶解，配成碱溶液。将银氨溶液在搅拌下逐渐加入到碱溶液中，再充分搅拌8h后，离心过滤出氧化银，用水洗涤3次，再用乙醇洗涤3次，在温度为80℃真空干燥，即得纳米级氧化银。本实施例所生产的纳米级氧化银平均粒径为82nm，最大粒径与最小粒径之差≤5nm。

实施例4

将硝酸银50Kg溶于300L水中，在搅拌下逐渐加入浓氨水400L，配成银氨溶液。另将固体氢氧化钠40Kg和1.0Kg保护剂（聚乙烯吡咯烷酮，分子量为30000）加水500L，搅拌溶解，配成碱溶液。将银氨溶液在搅拌下逐渐加入到碱溶液中，再充分搅拌8h后，离心过滤出氧化银，用水洗涤3次，再用乙醇洗涤3次，在温度为80℃真空干燥，即得纳米级氧化银。本实施例所生产的纳米级氧化银平均粒

径为 80nm，最大粒径与最小粒径之差≤5nm。

实施例 5

将硝酸银 50Kg 溶于 300L 水中，在搅拌下逐渐加入浓氨水 100L，配成银氨溶液。另将固体氢氧化钠 15Kg 和 0.2Kg 保护剂(聚乙烯醇，简称 PVA 分子量为 40000)加水 500L，搅拌溶解，配成碱溶液。将银氨溶液在搅拌下逐渐加入到碱溶液中，再充分搅拌 8h 后，离心过滤出氧化银，用水洗涤 3 次，再用乙醇洗涤 3 次，在温度为 80℃真空干燥，即得纳米级氧化银。本实施例所生产的纳米级氧化银平均粒径为 92nm，最大粒径与最小粒径之差≤5nm。

[19] Intellectual Property Bureau of the Peoples' Republic of China

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Patent Claims 1 page; Descriptions 3 pages

[54] Name of Invention: Nanometer Grade Silver Oxide and the Process for Its Manufacturing

[57] Summary

A nanometer grade silver oxide; the average particle size of which is 80 – 100 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm. The process of preparation is to dissolve silver nitrate in water, gradually adding concentrated ammonia water while stirring, to obtain an ammoniacal silver solution. Solid sodium hydroxide and a protective agent are then dissolved separately in water while stirring, to obtain an alkali solution. The protective agent may be polyvinyl-pyrrolidone or polyvinyl alcohol. The ammoniacal silver solution is gradually added to the alkali solution while stirring; after further through stirring, the solution is centrifuged to separate the silver oxide precipitate, which is then washed and vacuum dried; obtaining a nanometer grade silver oxide, with good stability and dispersibility, as well as uniformly distributed particle size. The process may be used for industrial manufacturing.

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Patent Claims

1. A nanometer grade silver oxide, the characteristics of which are that the average particle size is 80 – 100 mm; the difference between the largest particle size and the smallest particle size is $\leq$ 5 nm.

2. A process for manufacturing the nanometer grade silver oxide as mentioned in Claim 1, the characteristic of which are that an aqueous solution of silver nitrate is prepared, followed by the addition of ammonia water to obtain a ammoniacal silver solution, which is then gradually added to a sodium hydroxide solution with pre-added protective agent, forming super fine silver oxide precipitates; after filtration, washing, and drying, super fine silver oxide with an average particle size of 80 – 100 nm is obtained; the difference between the largest particle size and the smallest particle size is $\leq$ 5 nm; the protective agent mentioned is polyvinyl-pyrrolidone or polyvinyl alcohol.

Descriptions

Nanometer Grade Silver Oxide and the Process for Its Preparation

This invention is pertaining to nanometer grade silver oxide and the process for its preparation.

Silver oxide (Ag_2O) has been widely used in the industry for electronic components, button batteries, preservatives or antiseptics, cleaning agents, glass coloring agent and polishing agent, as well as chemical catalysts, etc.. Thirty percent of industrial silver usage is for production of silver oxide (or silver nitrate), in which the silver oxide consumption by the electronic component industry accounts for 90 % of the total silver oxide. Currently, domestic silver oxide are primarily prepared from the reaction of a silver nitrate solution with a sodium hydroxide solution to obtain a silver oxide precipitate, which is then washed, separated, and dried to obtain the silver oxide finish product. The major technical indexes (indicators) of the product include silver oxide content, clarity test and nitric acid insoluble materials, free alkali, nitrate salts, hydrochloric acid un-precipitated material content, and drying weight loss, etc..

The major existing issues include:

1. There is no particle size index in current product; the particle size is difficult to meet the requirements of various industries. The silver oxide used in the electronic component industry is usually prepared into a silver oxide paste, which requires extended ball milling levigation to reduce the size (require approximately 7 days) before mixing with other materials, which is then printed onto a device substrate (chip) through silk screening. The particle size of silver oxide is directly impacting the quality of the prepared silver paste and the cost of silk screen, which is in turn affecting the quality of the device. The requirement of the silver oxide particle size for the electronic industry is "the smaller the better". Current manufacturing technologies used both domestically and abroad are difficult to meet these requirements. Silver oxide used in the industries for chemical catalyst, medical antiseptics, glass coloring, etc., also requires a particle size of "the smaller the better".

2. Although domestically produced silver oxide is meeting the national purity standard, however, due to current processing technology, the particle size of current product is too large, making it easy to enclose impurities; it is very difficult to obtain high or super high purity grade silver oxide.

The objective of this invention is to provide a nanometer grade silver oxide, with good stability and dispersibility, and a uniformly distributed particle size, as well as a process for industrial manufacturing.

Technical thinking behind this invention: When a silver nitrate solution is reacting with a sodium hydroxide solution, the Ag^+ ion concentration in the solution is usually too high, the rate of growth of the silver oxide particle produced in the reaction system is too fast; at the same time, it is very easy to form aggregates between the particles, as a result, the particle size of the silver oxide produced is too large. If the Ag^+ ion concentration can be reduced during reaction, allowing the formation of silver oxide to speed up while the growth rate to slow down; at the same time, if the silver oxide particles are protected once they are formed, to prevent aggregate from forming, then, a silver oxide product with very small particle size can be prepared.

Therefore, the technical schemes of this invention are as follows:

A nanometer grade silver oxide, with an average particle size of ≤100 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

A process for manufacturing the nanometer grade silver oxide, which involves the preparation of an aqueous solution of silver nitrate, concentrated ammonia water is added to obtain an ammoniacal silver solution, which is then gradually added to a sodium hydroxide solution with pre-added protective agent, forming super fine silver oxide precipitates; after filtration, washing, and drying, super fine silver oxide with an average particle size of 80 – 100 nm is obtained;

the protective agent mentioned above may be polyvinyl-pyrrolidone or polyvinyl alcohol.

The specific process involves:

Dissolve 50 – 100 (parts by weight, same hereafter) of silver nitrate in 300 parts of water, add 100 – 400 parts of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 parts of water is added to 20 – 40 parts of solid sodium hydroxide and 0.2 – 1.0 parts of protective agent; it is stirred until dissolved to obtain an alkali solution. The protective agent may either be polyvinyl-pyrrolidone or polyvinyl alcohol. The ammoniacal silver solution mentioned above is gradually added to the alkali solution mentioned above while stirring. After the solution is further thoroughly stirred for 4 – 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared by the method of this invention has a particle size of 80 – 100 nm, has good stability and dispersibility, with a uniform particle size distribution.

Using the manufacturing process of this invention, the silver oxide obtained has an average particle size of less than 100 nm, the difference between the largest particle size and the smallest particle size is ≤ 5 nm; since a protective agent is being used, it has very good stability and dispersibility.

Application Example 1

Dissolve 50 Kg of silver nitrate in 300 L of water, add 100 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 40 Kg of solid sodium hydroxide and 0.2 Kg of protective agent (polyvinyl-pyrrolidone, or PVP, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 4 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 88 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 2

Dissolve 100 Kg of silver nitrate in 300 L of water, add 100 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water id added to 25 Kg of solid sodium hydroxide and 1.0 Kg of protective agent (polyvinyl-pyrrolidone, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 100 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 3

Dissolve 100 Kg of silver nitrate in 300 L of water, add 400 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 40 Kg of solid sodium hydroxide and 1.0 Kg of protective agent (polyvinyl-pyrrolidone, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 82 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 4

Dissolve 50 Kg of silver nitrate in 300 L of water, add 400 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 40 Kg of solid sodium hydroxide and 1.0 Kg of protective agent (polyvinyl-pyrrolidone, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 80 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.

Application Example 5

Dissolve 50 Kg of silver nitrate in 300 L of water, add 100 L of concentrated ammonia water gradually while stirring to obtain an ammoniacal silver solution. Separately, 500 L of water is added to 15 Kg of solid sodium hydroxide and 0.2 Kg of protective agent (polyvinyl-pyrrolidone, or PVP, molecular weight 30,000); it is stirred until dissolved to obtain an alkali solution. The ammoniacal silver solution is gradually added to the alkali solution while stirring. After the solution is further thoroughly stirred for 8 hours, the silver oxide precipitate is separated through centrifugation; the silver oxide is then washed with water for 3 times, followed by washing with ethanol for 3 times, and is dried under vacuum at a temperature below 80°C, to obtain a nanometer grade silver oxide. The nanometer grade silver oxide prepared in this application example has an average particle size of 92 nm; the difference between the largest particle size and the smallest particle size is ≤ 5 nm.